

Effect of Relative Humidity on Cement Paste: Experimental Assessment and Numerical Modelling



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Abstract In this study, the objective was to verify whether or not the classical Richard-Fick model was able to take into account the size effect on drying. First, mass loss experiments are made on ordinary cement paste cylinders of 3.6×18 cm geometry that enable us to calibrate the model parameters. Second, the quality of identification and drying model was checked by predicting mass loss evolution of small prism of $1 \times 5 \times 10$ mm size dried at different steps of relative humidity. The results demonstrate that the present drying model is able to predict the drying of specimen for different sizes and levels of humidity at ambient temperature. And finally, drying shrinkage experiments for different rates of drying are performed on cement paste cylinders of 3.6×18 cm; then, the prediction of drying shrinkage evolution was made using a simple model where the drying shrinkage is supposed to be linear with respect to the relative humidity. The result appears to be satisfactory. Since the spatio-temporal evolution of water content is needed as input to shrinkage prediction, the latter result confirms that the identification of drying model parameters is trustworthy.

Keywords Drying · Drying rate · Mass loss · Drying shrinkage

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1 Introduction

On the one hand, drying of cement based materials is a key factor of durability of concrete structures [1], therefore one needs to know its evolution on the long term for such structures [2]. On the other hand, drying is a very slow diffusion process, and may last for many decades for large structures such as large bridges or nuclear power plants [3]. Thus a modeling is needed to bridge that gap. Usually, models are calibrated on laboratory specimens [4] and then the parameters are used for prediction at structural level. Yet it is important to verify whether or not, this model accounts correctly for size effects. In this study, mass loss and sorption isotherm experiments have been conducted on ordinary cement paste specimens, for different sizes and at different rates of drying. Drying shrinkage measurement is also performed for some specimens. A drying model accounting for water permeation and vapor diffusion, [5] called Richards-Fick model in this paper is used to simulate the experiments. This model is calibrated by inverse analysis and then used for prediction. The prediction is shown to be very satisfactory. Therefore, we were confident to predict drying shrinkage. By using the well-known drying shrinkage kh model proposed by [6, 7], we were able to reproduce correctly the drying shrinkage at different drying rates with a single set of parameters. First the method of study is introduced. Second the experiment and simulation results are presented. Then the discussion on the results is undertaken on the following section. And last, we end this paper with conclusion on the whole study.

2 Method

2.1 Experiments

The material we studied is a cement paste, of 0.525 w/c ratio. A cement of type CEM I 52.5 N is used, corresponding to the VERCORS concrete, [8, 9]. All specimen are kept under endogenous conditions just after casting up to testing date. Below are listed the different experiments of the study:

- Test CP1: Sorption isotherm of the material is characterized first by means of dynamic vapor sorption (DVS).
- Test CP2: Mass loss measurement is made on specimen of $1 \times 5 \times 10$ mm geometry using the same DVS used to test CP1
- Test CP3: Mass loss and drying shrinkage tests are performed on cylinders of 3.6×18 cm size, for three humidity levels obtained with saline salt solutions, embedded in specific designed chambers.
- Test CP4: A final test is made in testing room on cylinder of 3.6×18 cm size, where humidity and temperature are kept constant (50% and 20 °C).

2.2 Modelling

Drying model: The drying model used herein was implemented in code aster (<https://www.code-aster.org>). A non-linear diffusion equation governs the evolution of liquid water saturation degree S , modeled by “Eq. (1)”. The equivalent coefficient of diffusion D in “Eq. (2)” takes into account for permeation of liquid and diffusion of water vapor in the porous network of the material.

$$\frac{\partial S}{\partial t} = \nabla(D(S)\nabla S) \quad (1)$$

$$D = \frac{\partial P_c}{\partial S} \left[\frac{K_0 k_{rl}(S)}{\emptyset \mu_l \left(1 - \frac{\rho_v}{\rho_l}\right)} + D_{v0} d(S) \left(\frac{M_v}{\rho_l R T}\right)^2 P_{vsat} e^{\left(\frac{P_c M_v}{\rho_l R T}\right)} \right] \quad (2)$$

$$P_c = a(S^{-b} - 1)^{1-\frac{1}{b}} \quad (3)$$

$$k_{rl} = S^{n_k} \left(1 - (1 - S^b)^{\frac{1}{b}}\right)^2 \quad (4)$$

$$D_v(s) = D_{v0} \emptyset^{a_{mq}} (1 - S)^{b_{mq}} \quad (5)$$

where:

- S is the liquid water saturation degree, chosen herein as the internal variable of the model
- K_0 is the intrinsic permeability of the material to be identified
- P_c is the capillary pressure which can be related to saturation degree using Van Genuchten [10] two parameters relation and reads “Eq. (3)”; where b is the same as in k_{rl} relation, and a , a parameter to be identified on desorption isotherm of the given material.
- k_{rl} is the relative permeability of the material, which depends on saturation degree. A choice is made here to use Mualem empirical relation “Eq. (4)” [10], where n_k is a material parameter to be identified.
- $D_v(s)$ is the vapor diffusion coefficient of the material, it decreases when the water content increases. It is chosen here to use Mualem [10] equation shown in “Eq. (5)”; where D_{v0} is the diffusion coefficient of the material at dry state; a_{mq} , b_{mq} are the parameters describing the tortuosity of the material and are to be identified by inverse analysis on mass loss experiment.
- \emptyset is the porosity of the material measured experimentally,
- ρ_l , ρ_v , M_v are, respectively, the bulk density of liquid water, the bulk density of vapor, the molar volume of vapor.
- T is temperature, considered to be constant in this modelling and R is the universal gas constant.

- The boundary condition adopted here is of Robin type and reads “Eq. 6” where S_e is the equivalent saturation degree of water in air close to drying surface, S_i the saturation degree at drying surface level and C_s the surface exchange factor to be identified.

$$f_s = C_s(S_e - S_i) \tag{6}$$

Drying shrinkage:

The drying shrinkage is supposed to be proportional on the humidity change rate [7]:

$$\dot{\epsilon} = k\dot{h} \tag{7}$$

2.3 Identification Method

Drying model First parameters a and b of capillary pressure in “Eq. (3)” are identified using sorption isotherm measurement (Test CP1, see “Fig. 1”). The calibration of the isotherm is very satisfactory.

Second parameters K_0 and n_k describing liquid water permeation in porous media are determined using Test CP4, and Dirichlet boundary condition, see “Fig. 2”.

And last, the parameters a_{mq} accounting for tortuosity of the material and the surface factor C_s accounting for real boundary condition are fixed by performing optimization on mass loss measurement of Test CP3-1. Experimental results of CP3-2, and CP3-3 are only used in aim of prediction, see “Fig. 3”.

Fig. 1 Fitting experimental isotherm of Test CP1 of cement paste w/c = 0.525 by Van Genuchten model

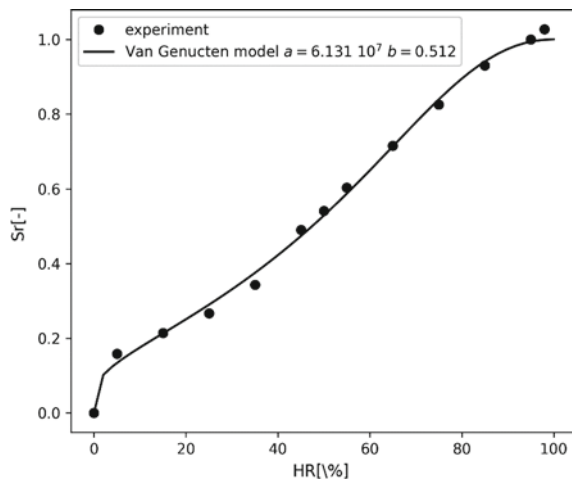


Fig. 2 Identification of intrinsic water permeability $K_0=1.02 \cdot 10^{-21}$ and the relative permeability parameter $n_k = 2.52$ on experiment Test CP4

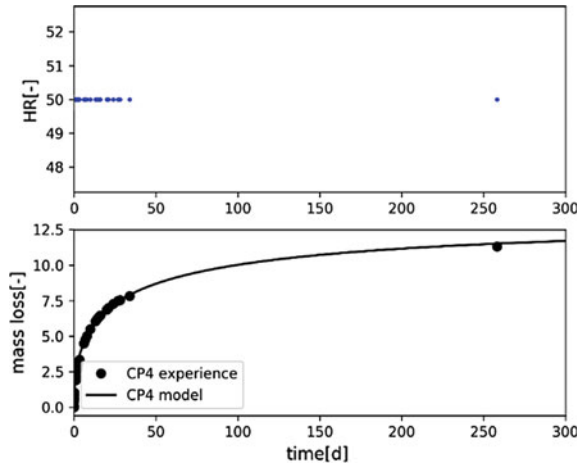
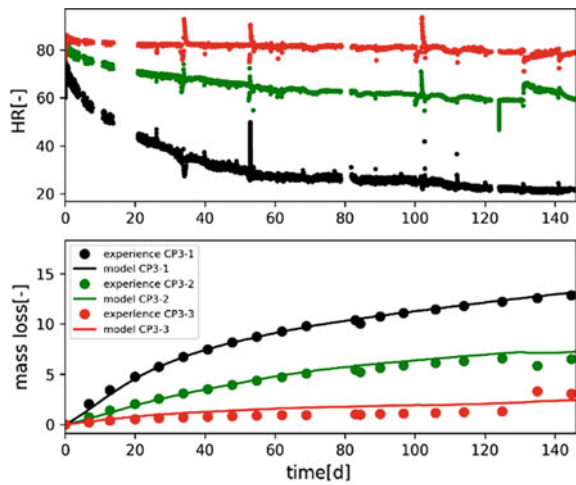


Fig. 3 Identification of a_{mq} and C_s on mass loss measurement performed on cement paste $W/C = 0.525$, cylinders of 3.6×18 cm



As a further validation of the drying model identification shown on “Fig. 3” following the procedure described above, let us now show that the model is able to reproduce the mass loss evolution of the very thin specimen ($1 \times 5 \times 10$ mm) used in Test CP2, except for the last humidity step of 5% RH. The results are shown on “Fig. 4” where red curve corresponds to simulations results with Robin type boundary condition, (the surface factor used herein is the one identified above) and the black curve is what is obtained using Dirichlet type boundary conditions.

Drying shrinkage: To simulate contraction caused by drying, a full water transport analysis is necessary. A spatio-temporal evolution of water obtained, thanks to the present calibrated Richards-Fick model is used as input for shrinkage model calibration. Then only one parameter has to be identified for all specimen. It is identified

Fig. 4 Prediction of mass loss of small prism ($1 \times 5 \times 10$ mm) of 1 mm drying thickness performed by DVS (Test CP2)

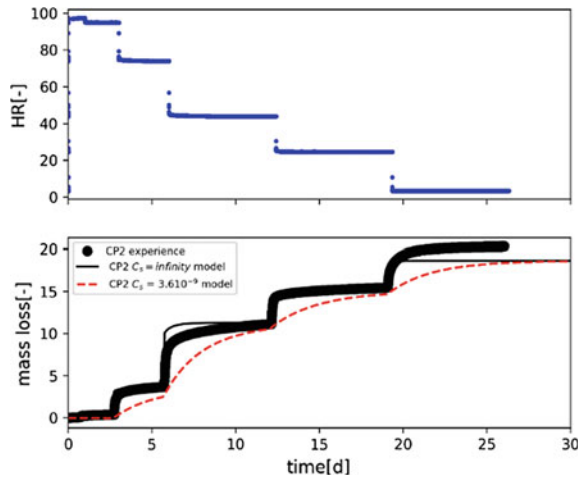


Table 1 Identified parameters of the drying and drying shrinkage models

$K[m^2]$	$n_k[-]$	$a_{mq}[-]$	$C_s[Kg.m^{-2}.s^{-1}]$	$k[-]$
1.0210^{-21}	2.52	5	3.610^{-9}	0.008

Table 2 Fixed parameters of drying model for the studied material

$\varnothing[-]$	$\rho_b[Kg.m^{-3}]^a$	$S_{init}[-]^b$	$b_{mq}[-]$	$P_{vsat}[Pa]$	$\mu_l[Pa.s]$	$\rho_v[Kg.m^{-3}]$	$T[K]$	$R[Kg.m^2.s^{-1}]$
0.465	1870	0.99	4.2	3062	0.001	0.018	293	8.314

^aBulk density of cement paste

^bInitial saturation for all studied specimen

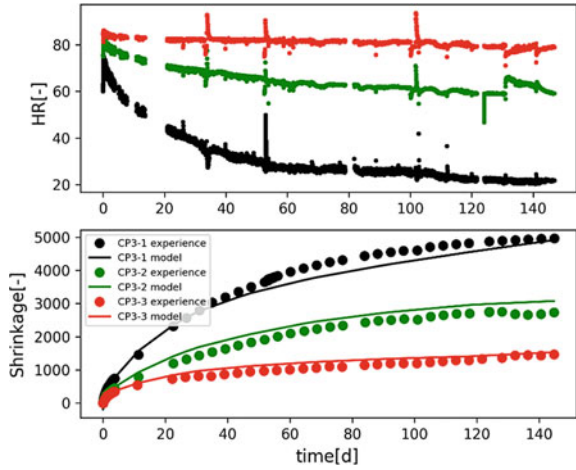
on test CP3. All the parameters are displayed on “Table 1” for identified parameters, and “Table 2” for fixed ones. The temperature was kept constant in all experiments, close to 20 °C and the relative humidity condition for each test is displayed on the corresponding figures.

Once the shrinkage parameter identified, the agreement with the experimental results is satisfactory for all drying rates, ref “Fig. 5”.

3 Discussion

The Richards-Fick model parameters have been identified successfully on various experimental tests performed on specimens of different geometry, size and level of drying humidity. Usually the model is used for relative humidity above 50%, where the contribution of vapor to drying process might be neglected [5]. For the present study, it is shown that the model can be used to predict mass loss up to 20%

Fig. 5 Identification of drying shrinkage model described above, using Test CP3 1, 2, 3, $k = 0.008$



relative humidity tests by taking into account the vapor diffusion contribution in the modeling. A question may raise to know whether the surface factor C_s incorporated for identification cannot impede the identified parameters. To ensure that it is not the case, we identified the contribution of liquid water permeation parameters (K_0 , n_k) on Test CP4, performed on vast room regulated on temperature and humidity, enough to allow instantaneous exchange between the exposed drying surface of specimen, with its environment. But in case of Test CP3, the exchange speed at the specimen surface with atmosphere, was very low, because of use of hermetic chamber without any ventilation. It was then necessary to introduce surface factor on boundary conditions, to reproduce the experiments. The prediction made on Test CP2, Fig. 4 demonstrate that C_s is specific to Test CP3 and the drying material parameters identified herein are intrinsic to the material. The calibrated model was able to predict correctly the mass loss evolution of specimen of $36\times$ smaller size, for different steps of relative humidity ranging from 98% up to 20% RH. For the step of 5% RH, the model underestimates the mass loss. Another good surprise, is that we noticed that the simplified drying shrinkage model based on the proportionality between drying contraction and humidity change rate was able to predict the experiment results for different drying rates, and for humidity below 50% relative humidity. We think that it was made possible thanks to use of real sorption isotherm of the material. This point will be investigated further by testing other models.

4 Conclusion

In this study, Richards-Fick model is tested against different sets of experimental data, and the identification of the model is shown trustworthy, since the identified parameters are calibrated and allow to predict mass loss measurement of specimen of

different geometry, size and drying conditions. The drying model was able to simulate drying condition down to 20% RH at ambient temperature, and assuming the drying surface exchange factor C_s to be infinite, is quite realistic, if air ventilation at the drying surface is speed enough to allow removing instantaneously the water vapor at the specimen surface. It was also found that if the desorption isotherm of the material is measured and calibrated properly, and if the Richards-Fick drying model to get a realistic moisture distribution inside the specimen, the phenomenological drying shrinkage model $\dot{\varepsilon} = k\dot{h}$, can predict correctly the strain evolution, for different drying rates and for relative humidity up to 20% at ambient temperature. More investigation on drying shrinkage, both for experimental and numerical point of view is undergoing now, and the results will be available soon.

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